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Re: Pilot Scale Conversion of Estevan Lignite
Into Hydrocarbon Distillate by Hydroprocessing

Dear Sir:

We are pleased to enclose 10 copies of the report of the Pilot Scale Conversion of Estevan Lignite into Hydrocarbon Distillate by Hydroprocessing. Under this contract we produced 25 litres of lignite-derived distilled fraction -350°C which were shipped to your laboratory. The report contains a complete summary of the work and results obtained, as well as the analysis of the material produced.

We would be pleased to review the report at your convenience and thank you for your continued confidence in our work. We shall be glad to continue to provide you with research services in the future and would appreciate greatly knowing how the fractions we produced, performed in the subsequent treatment.

Yours sincerely,

G. Teodosiu
Project Manager

GT:vb

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1.0 INTRODUCTION

SANDWELL TECHNOLOGIES undertook this contract at CANMET's request in order to produce a quantity of homogeneous coal-derived distillate material to be used in the hydrotreating catalysts development work at CANMET Energy Research Laboratories.

Sandwell Technologies has dedicated its efforts in the last seven years to work in direct hydroliquefaction of Western Canadian coals. Through shared funding by Sandwell & Company and EMR, a 0.1 TPD process development unit was designed, commissioned and operated at the Mississauga research facilites of Sandwell Technologies. This unit has been in operation since 1981 for testing of various coals and process development studies providing Sandwell Technologies with a unique data base on low rank coal liquefaction.

The work for this contract was carried out in the existing equipment using a shipment of lignite received in December 1984 from the Manalta Coal Company and ground at the Mineral Processing Section of the Ontario Research Foundation.

25 Kg of distillate material were produced through processing of 75 Kg of lignite using a start-up solvent prepared by Sandwell Technologies in previous runs.

Sandwell Technologies welcomed this opportunity of participating in the overall coal liquefaction effort in Canada.

2.0 SUMMARY

The work described in this report consisted in the production of 25 litres of coal-derived distillate from Estevan lignite, by hydroliquefaction. The report includes a description of the equipment and procedures used, a summary of the PDU operation, data acquistion and the material balance. Also included are the characterization of the lignite coal, start-up solvent and of the final product.

SOMMAIRE

Le travail qui fait l'object de cet raport a eut comme but la production d'une quantité de 25 litres de produit distillé a partir de la lignite de Estevan par hydroliquefaction.
Le raport contient la déscription de l'equipment et des procédures utilisées aussi que un sommaire des opération de l'unite pilot, l'aquisition de données et un bilan des matériaux.
Le raport inclue aussi l'analyse chimique du charbon, solvent de démarrage et celle du produit final.

2.1 PDU OPERATION

For the production of the distillate as required in the contract, a continuous run was carried out on the 0.1 PDU in recycle mode at 410°C reactor temperature and 24 MPa pressure with hydrogen. About 75 Kg. of fresh coal was fed into the unit.

The start-up solvent was lignite-derived oil fraction 230-450°C from Sandwell's stock of previously produced material. During the run, the product was vacuum distilled for the recovery of the solvent fraction.

The -250°C fraction was accumulated and was shipped to CANMET as Naphtha fraction. At the conclusion of the run, 50 Kg of heavy product was vacuum distilled and 21 Kg of the -350° fraction was shipped to CANMET. The remaining product was distilled for production of start up solvent to be returned to the standby stock.

L'OPÉRATION DE L'INSTALLATION PILOTE

Pour la production du matériel specifie par le contract, l'installation de 0.1 tonne par jour de SANDWELL TECHNOLOGIES fonctionna avec récyclage du solvent à une température du réacteur de 410 degrees Centigrades et une pression de 24.2 MPa avec de l'hydrogéne pur, a une vitesse spatiale horaire du liquide de 1.0 h^{-1}. Environs de 75 Kg de charbon ont été introduits dans le réacteur a un débit horaire de 5-6 Kg/h.
Le solvent de démarrage fut une fraction 230-450 deg.C produit part SANDWELL TECHNOLOGIES dans une opération antérieure. Pendant la marche de l'unite le solvent de récyclage fut recouperé par distillation sous vide du produit. La fraction légére (-250 deg.C) fut accumulée et fut livre a CANMET comme fraction "naphtha". Aprés la fin de l'operation 50 Kg de produit lourd furent distillés sous vide. Une quantité de 21 litres de la fraction distillée moins de 350 deg.C furent livres à CANMET. Le rest du produit fut distillé pour future utilisation comme solvent de démarrage.

2.2 RESULTS

The material balance closure for this run was 97.2%. The coal conversion was 75% wt. maf, lower than previously obtained in our runs with Estevan lignite - this fact could be attributed to the lower reactor temperature applied in this run or the fact that the new lot of lignite may have been less reactive.

An investigative batch test conducted prior to the run also resulted in low conversion, which points to the lower reactivity of this coal shipment.

The liquid yield was also lower - only 51.5% wt. maf. The distillate fractions produced for CANMET had a density at 21°C of

0.9335 for the -250°C fraction
1.0335 for the -350°C fraction

The simulated distillation results are shown in Section 5 of the report and showed that 80 - 90% of the material, boiled in the desired range, with the balance boiling up to 470°C.

The elemental composition indicated good oxygen removal but a fairly low hydrogen content confirming that in the thermal liquefaction step at the applied conditions, most hydrogen consummed goes into gas and water making.

RÉSULTATS

La bilan des matériaux calculé pour cette operation fut de 97%. La conversion du charbon obtenue fut moins que celle obtenue dans des marches antérieures avec lignite de Estevan. Cet resultat peut être attribue à la température du reacteur que fut plus basse ou peut être que le dérnier lot de charbon fut moins reactif. Le rendement en liquide obtenue fut aussi plus bas (51.5% maf) que celui obtenu dans les marches anterieures.

La qualitee de fractions distillées prepares pour CANMET a ete verifie par l'analyse de:

- densite a 21 deg.C
  0.9335 pour -250 fr. et
  1.0335 pour -350 fr.
- distillation simulée (les courbes sont montrées dans la section 5 du raport) indiqua que de 80 à 90% du matériel se trouve dans l'interval desiré et le rest distille jusqua 470 deg.C.
- l'analyse elémentaire indique que une bonne deoxygenation a a eut lieu, par contre le contenu relativement bas en hydrogéne confirme que dans la liquefaction termique de la lignite la plus part de l'hydrogene est dépensé pour produire des hydrocarbures gaseuses et de l'eau.

3.0 DESCRIPTION OF EQUIPMENT PROCEDURES

3.1 DESCRIPTION OF UNIT MODULES

A simplified chart of the continuous flow system utilized for the production of distilled from lignite by hydroliquefaction is shown in Figure 3.1 The system can be regarded as a combination of seven modules,

- Slurry preparation and feed,
- Fresh gas manifolds,
- Reactor,
- Pressure let-down and product collection,
- Gas scrubbing,
- Gas recirculation,
- Solvent recovery-Vacuum Distillation Unit.

The gas feed-lines, storage and compressor are installed in a different room separate from the rest of the system.

The Slurry Preparation and Feed Module is composed of the slurry preparation and the slurry feed vessels. The slurry preparation is done in a 50 L tank provided with a fast propeller-type stirrer, electric heater, recirculation pump and connection to inert gas and to the slurry feed tank. The loading of this tank is made batch-wise, each batch being enough for 8 hours unit operation at a feeding rate of 6 kg of slurry per hour. From this vessel the slurry is transferred to the slurry feed tank. The construction of this tank is similar to that of the preparation tank but for the purpose of feed rate monitoring, this vessel is suspended on a load cell. The slurry is continuously recirculated by a low-pressure centrifugal pump at a sufficient rate to keep the coal in suspension. The slury is heated under inert gas blanket at all times. A high pressure piston pump takes suction from the recirculation line at a pressure of 50 - 70 KPa. The slurry feed rate can be changed by adjusting the piston stroke.

Two such high pressure piston pumps are installed in parallel:

- Bran and Lubbe model No. N31
- Milton Roy Simplex Model B (MRI-84-92).

The Fresh Gas Module includes a separate manifold for each of the following gases: hydrogen, carbon monoxide, nitrogen and helium; each provided with pressure regulators. The hydrogen and carbon monoxide manifolds are made of 1/4" slim line 316 SS tubing. Each cylinder is connected by a brass coil to an arrangement of one check valve, one globe valve and a tee fitting to the manifold. There is one pressure gauge for each manifold and one pressure regulator to ensure constant feed pressure to the compressor. The hydrogen and carbon monoxide manifolds have 6 ports (3 cylinders in use, 3 full in wait) and the inert gas manifold has 3 ports.

The fresh gas is supplied from commercially available cylinders.

The flow of each individual gas is regulated by a micrometric valve.

The Reactor Module is comprised of the slurry preheater, the gas preheater, the upflow the reactor, the hot high-pressure receiver, the cold high-pressure receiver, water condenser and the back pressure regulator.

The slurry preheater is made of a 20 ft. coil of high pressure, 316 SS tubing 3/8" imbedded in a heat transfer cement block, heated by an electric heater. The gas preheater is a 1" diameter pipe, provided with strip heaters. Temperatures in both preheaters are regulated from the temperature of the fluid leaving the preheater. The provision of introducing a split gas flow before and/or after the preheater is a built-in option to introduce cold hydrogen serving a double purpose: to prevent coking the slurry preheater in case of a temperature-run in the preheater and to help cool the in-flowing slurry in either the preheater or the reactor in case of a temperature run in the reactor.

The reactor is a 6 cm diameter, 215 cm long 316 SS tubular upflow reactor, built with modified high-pressure Bridgeman closures and provided with four heating zones, individually controlled from thermocouples placed directly in the flow. The outside wall temperature is also monitored and controlled.

The high temperature, high-pressure receiver (M3X1) where the first flash takes place is a 7.5 cm diameter by 86 cm (316 SS) and it is provided with the same type of closures as the reactor. The liquid level is kept constant by a gamma ray level indicator which controls the operation of pressure let-down valves.

The vapour phase is cooled to around $20^{o}C$ in a water jacketed receiver (M3X2) which is 8 cm diameter by 80 cm flanged vessel, (316 SS). The liquid condensed in vessel M3X2 is collected in a 300 ml intermediate vessel (316 SS) and evacuated periodically through receiver (M3X3) using a manual let-down procedure. All high pressure vessels have been designed to withstand a working pressure of 35 MPa at $450^{o}C$.

The liquid phase from the hot high-pressure receiver is evacuated via the pressure let-down module. The heavy product pressure let-down module is constituted of two separated trains, each comprising two sequentially operated Badger Meter Research valves, with Tungsten Carbide seats and stems, actuated on/off, with a 50 cm^3 intermediate vessel in between. The timing of the valves is manually adjusted, based on the indication of the gamma-ray indicator, the feeding rate and reaction parameters.

The slurry stream can be directed to either of the let-down trains while the other one is completely isolated allowing eventual repair work to proceed.

The heavy liquid stream off the let-down system is collected in a 15 L, electrically heated, hot receiver (M4X5) operating at max. 690 KPa where the

last of the dissolved gas, water vapors and products boiling lower than 200°C are vaporized in the process of the pressure release, and are condensed in a water cooled condenser to be collected as intermediate product in the low pressure, low temperature receiver (M4X6).

The Gas Scrubbing Module operates at ambient temperature and 2.8 MPa. The scrubber itself is a 10 cm diameter, 210 cm long, packed column where the gas moves counter-current to the absorbing medium which can be water, caustic solution or methanol. The absorbent solution is circulated with a pump to the top of the scrubber and its compositon is periodically analyzed. The solution leaving the scrubber through a bottom valve is kept in a closed tank connected to the exhaust system in order to allow the dissolved gases to escape.

The Gas Recycle Module involves a 10 L scrubbed gas storage tank (M6X1) designed for 7 MPa kept at maximum 4 MPa by a system of spring-loaded pressure regulators. A PPI diaphragm compressor takes suction from this tank and discharges the gas mixture at high-pressure into a 15 L buffer tank (M6X2) designed for 35 MPa which serves as a gas feed tank. The recycle gas composition is analyzed regularly and calculated rations of fresh make-up gases are added into the compressor's suction to achieve the desired H_2content.

The Vacuum Distillation Module consists of a batch operated still (20 L) equipped with a packed distillation column, electrically heated and connected to a vacuum pump via liquid-nitrogen-cooled traps.

3.2 TESTING PROCEDURE

Coal Slurry Preparation

The pre-weighed ground coal (as is) was slurried batch-wise, under nitrogen atmosphere with the solvent in the slurry preparation vessel M2X1. The slurry was well-mixed and heated to 60°C before it was transferred to the slurry feeding vessel M2X2. The slurry in M2X2 was maintained at 60°C under continuous stirring and recirculation.

Slurry Feed and Preheating

The coal slurry was fed into the unit using the high-pressure piston pump at the rate of 5 Kg/hr. It was preheated to a maximum of 300°C in the slurry preheater before entering the upflow reactor.

A portion of the hydrogen gas feed was added to the coal slurry, upstream of the preheater inlet.

Gas Feed and Preheating

Fresh hydrogen from the manifolds was supplied to the suction of the diaphragm compressor. The compressed gas was stored in the high pressure buffer tank

(M6X2) out of which it was supplied through a pressure regulator into the unit at 24.15 MPa. The flow rate of the gas into the unit was maintained around 1100 SL/hr and it was measured with a differential pressure gauge.

The inlet gas was heated to 350°C in the gas preheater before it reached the reactor.

Reaction Zone

The preheated coal-slurry and gas streams were fed into a mixing chamber situated below the upflow reactor. The temperature of the reactor was maintained at 410-420°C constant by controlling the four (individually controlled) outside wall temperatures.

The effluent of the reactor was cooled to around 200°C in the first separator.

High Pressure Gas and Slurry Separation

The vapour and slurry phases were separated in the High Pressure Separator (M3X1).

The uncondensed gas phase was vented to the scrubber through the back pressure regulator M3BPRV. The condensed light hydrocarbon and water collected in the 300 ml intermediate vessel was evacuated periodically through receiver M3X3 using a manual pressure let-down procedure.

The liquid slurry from M3X1 was continuously evacuated through the pressure let-down valves and was collected in the low pressure high temperature flash tank M4X5 which was emptied periodically and the product was weighed and analyzed for solids and water content. A portion of this product was further processed in the vacuum distillation unit to recover the recycle solvent fraction and a portion of it was used without further processing as recycle solvent.

The distillate 250-430°C fraction was combined with non-distilled heavy product and with fresh coal in the ratio of approximately 1:1:1 to prepare the feed for further operation.

The vapor phase flashed out of M4X5 was condensed in a water-cooled condenser and collected in the low pressure, low temperature receiver (M4X6) which was emptied. The product, containing most of the water produced, was weighed and the organic phase separated. This product was accummulated and at the end of the run it was distilled in order to produce the -350°C fraction.

Gas Recovery

The gas stream released from the back pressure regulator M3BPRV was passed through the scrubber. The hydrogen sulphide and most of the carbon monoxide were removed and the scrubbed gas was vented after being measured and analyzed chromatographically.

The other gases flashed out of M4X6, M5X1 and M3X3 were separately measured, analyzed chromatographically and vented into a bubbler filled with a solution of sodium hydroxide.

3.3 DATA ACQUISITION

Data regarding reaction parameters (pressure, temperature), feed and product flows were monitored continuously. Most stream flows were measured directly and a few are calculated, as shown in Figure (3.2).

Routine periodic analysis of most streams was performed in order to check the correct operation of the system.

Feed/product sampling and weighing procedure allows complete mass balance and product distribution calculation for any time period during the run. Weights and samples were taken throughout the run on a regular basis.

The main streams and sampling points are shown in Figure 3.2. A data acquisition schedule is given in Table 3.1, outlining sample analysis and frequency of analysis as well as the type of weight or flow measurement.

Feedstock

The coal was sampled from different areas in a 200 kg drum and a composite sample was analyzed. The starter solvent was heated and well-mixed before a representative sample was taken for analysis.

Samples of the feed slurry were taken from the feed vessel M2X2. At least three samples were taken per batch in order to yield information on water loss from the feed vessel by evaporation and to double check the solids concentration.

The flow of the input gas was monitored continuously. The D/P cell and the integrator counter readings were recorded at regular intervals.

Products

The heavy product was sampled directly from the hot high pressure receiver (M3X1) during a let-down sequence and analyzed immediately for THF insolubles in order to follow the coal conversion evolution.

For a quick indication of the stability of overal reaction all products recovered from the unit were weighed and the weights recorded. The light product flow was visually separated into aqueous and organic phases.

Table 3.1 SANDWELL TECHNOLOGIES UNIT DATA ACQUISITION SCHEDULE

STREAM	COMPOSITION		FLOW	
	ANALYSIS	FREQUENCY	METHODS OF MEASUREMENT	FREQUENCY
Feedstock				
Coal	proximate ultimate	as required	spring scale (± .05 Kg)	each batch
Starter solvent	elemental distillation	as required	spring scale (± .05 Kg)	each batch
recycle solvent	% THF insolubles	each batch	spring scale (± .05 Kg)	each batch
gas in			D/P cell calibrate for each run (± 5%)	continuous
feed slurry	% THF insolubles % H_2O	at least 3 times/batch as required	strain gauge (± .1 Kg)	continuous manually recorded every 1/2 hour
Products				
heavy (M3X1)	% THF insolubles % H_2O	1 hour as required	not measured	
(M4X5)	% THF insolubles % H_2O	as required	spring scale (± .05 Kg)	1 hour
light (M4X6)	% H_2O % organics	as required	spring scale (± .05 Kg)	1 to 3 hours
Residue			spring scale	as required

Table 3.1 (continued) SANDWELL TECHNOLOGIES UNIT DATA ACQUISITION SCHEDULE

STREAM	COMPOSITION ANALYSIS	FREQUENCY	FLOW METHODS OF MEASUREMENT	FREQUENCY
light fraction	% H_2O % orgainc s	as required	spring scale ($\pm$ .05 Kg)	1 to 3 hours
Scrubbed gas	GC analysis for H_2, CO, CO_2 C_1, C_2, C_3, H_2S	as required	D/P cell calibrated for each run	continuous (integrated)
M3X3 and M4X5 off gases	GC analysis for H_2, CO, CO C_1, C_2, C_3, H_2S	as required	dry gas meter	continuous (readings periodic)
M5X1	GC analysis for H_2, CO, CO_2 C_1, C_2, C_3, H_2S	as required	wet testmeter	continuous (readings)

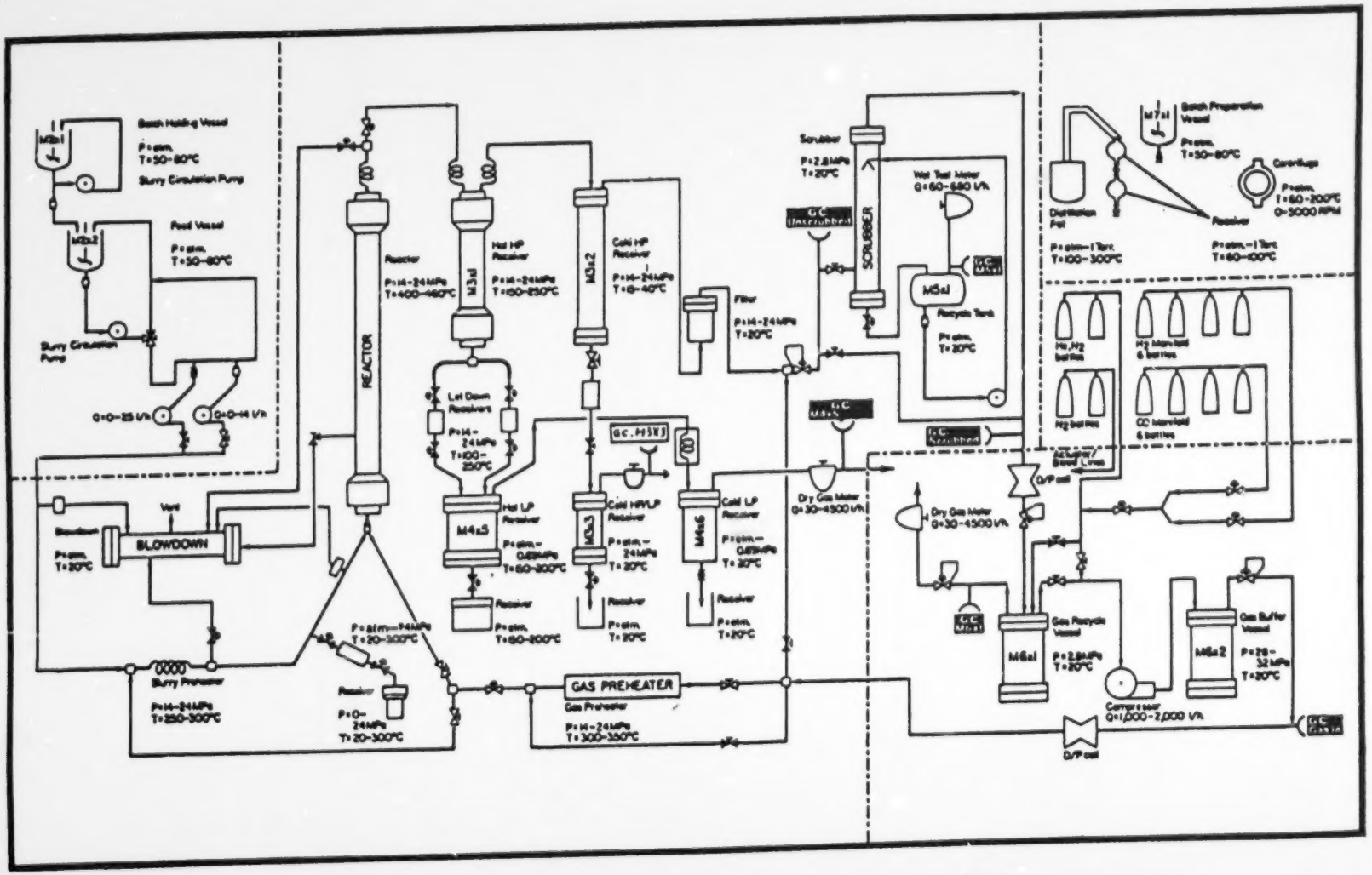

Figure 3.1 **SBRG Continuous Unit Flow Diagram: Volumes and Typical Operating Conditions**

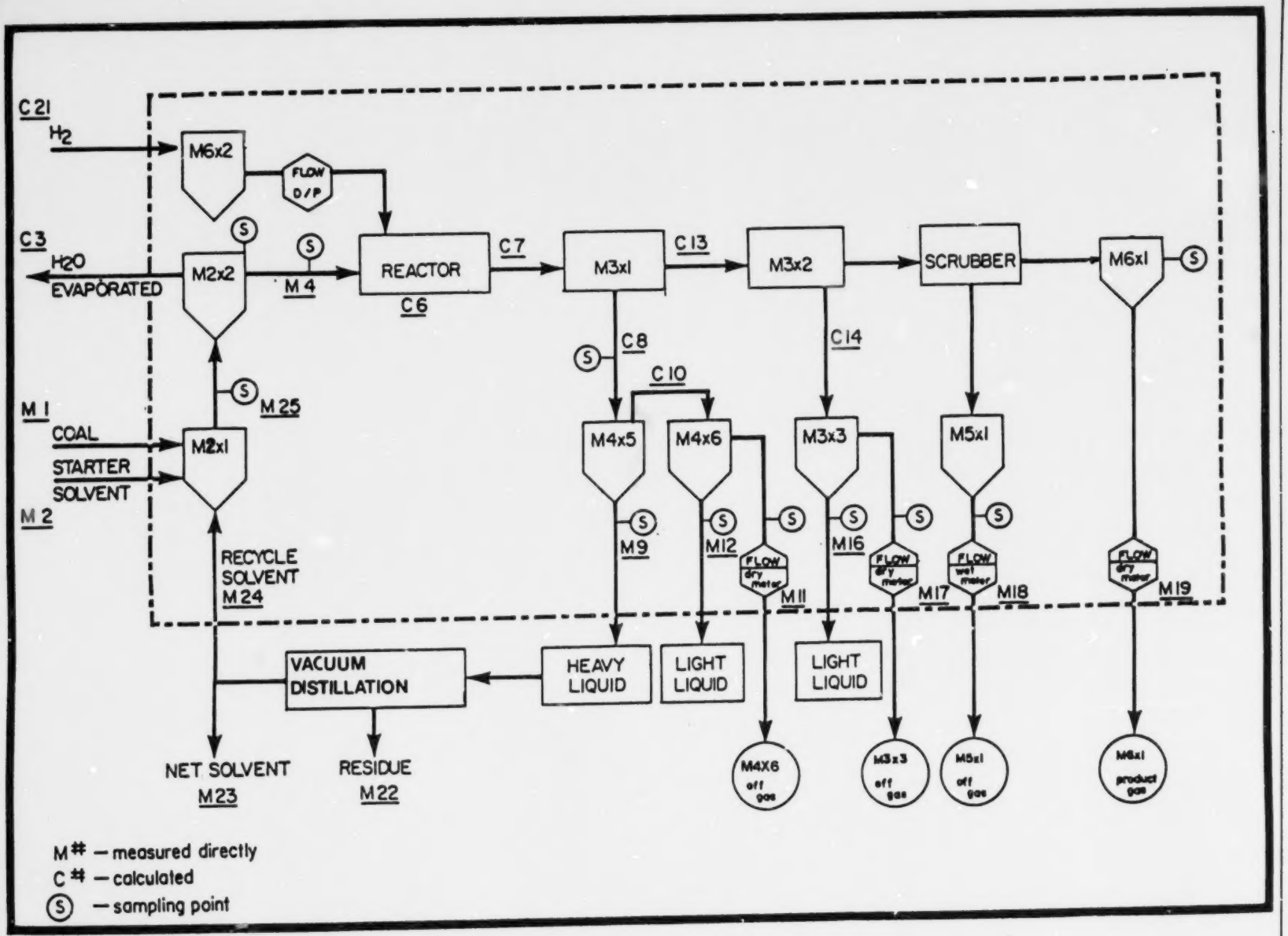

Figure 3.2 **SBRG Continuous Unit Data Acquisition**

Samples of the four gas outlets were taken regularly and analyzed on the gas chromatograph. The flows of these gases were measured on dry gas meters and wet test meters depending on pressures and flow rates.

A complete set of pressure, temperature and flow readings was taken at regular intervals and a run history of adjustments, samples, spills and equipment behaviour was maintained throughout the run.

4.0 ANALYTICAL PROCEDURES

4.1 SAMPLING TECHNIQUES

Most materials handled such as feed slurry, heavy product, solvents, light products and residues are heterogeneous, at least at room temperature. Consequently in order to obtain representative samples for analysis these materials must be thoroughly mixed and if required heated above 50°C before a sample was taken.

These samples are either used immediately or they are stored at -17C until use. In cases when the whole quantity of sample is not used for one analysis the heating and thorough mixing is repeated before aliquots are taken for specific analyses.

The light products which are generally present in two phases are separated into organic and aqueous phases before taking the samples for analysis.

Feed and heavy product samples during the run are obtained from process flow lines and considered representative. Samples obtained from storage containers are obtained after heating and sufficient mixing.

Several analyses performed on aliquots of the same sample or from samples taken at close intervals assure consistency in the overall sampling/analysis results.

4.2 METHODS OF ANALYSIS

4.2.1 Determination of Water Content

Determination of water content in the feed slurry and heavy product was done by Xylene azeotropic distillation (ATSMD 95-70).

The water in the light product was separated from the organic fraction using a separatory funnel and was measured volumetrically with a graduated cylinder.

4.2.2 Determination of Density

The density of the orgainc liquids was determined by the use of pycrometers at 21°C.

4.2.3 Insolubles by Filtration (for conversion calculations)

THF insoluble content of the heavy product was determined during the runs by filtration. The method yielded results within 20 minutes of sampling from the continuous unit.

Between 5 and 10 grams of sample were weighed in a beaker and 100 millilitres of THF were added to the beaker. The contents of the beaker were mixed and filtered through two previously weighed glass micropore filters (934-AM) under vacuum and on a Buchner funnel. The filters are then dried for 10 minutes at 100°C and weighed. The insoluble content is obtained by difference.

The method yielded precisions of ± 0.6%.

4.2.4 Simulated Distillation

The distillation curve of the organic liquids was obtained by subjecting representative homogeneous samples to simulated distillation on (ATSM D2887).

The simulated distillation system consists of the Perkin-Elmer Sigma-3 gas chromatograph with dual flame (FID) detectors and a Perkin-Elmer Sigma-10 data station. The point data for simulated distillation were derived from the calibration curve, using normal paraffins as the boiling point standard. The boiling points were calculated at 10 wt% intervals and include the initial and final boiling points, which correspond to 0.5. and 99.5 wt% points.

4.2.5 Elemental Analysis

The elemental composition was determined using a Perkin-Elmer 240 Elemental Analyzer wih simultaneous analysis of carbon, hydrogen and nitrogen in combustible matter.

The sulphur content was determined using a Leco 32 Sulphur Analyzer.

The oxygen was calculated by difference.

5.0 OPERATION AND RESULTS

A lot of .5 T of coal was obtained from Manalta Coal Company and it was pre-dried and ground by ORF to -100 mesh. The ground coal was delivered to Sandwell Technologies stored in 45 gal. drums lined with plastic and closed under CO_2 atmosphere.

75 Kg of the coal were actually used for this run. The run was started with a previously prepared coal-derived oil (distilled fraction 230-450°C), and after the reactor temperature reached 400°C, coal was introduced at a ratio of 1:2 coal to solvent. A mass balance calculation was carried out over 42 hours of operation.

The main system parameters were maintained constant during the run as shown in Table 5.1.

The slurry feed was prepared of fresh coal and recycle solvent in a ratio of 1:2. The recycle solvent was a mixture of distillate 230-420°C and heavy product as collected. The THF insoluble in the feed was maintained around 37% wt.

The operation was smooth, and the solvent recovery method used was by vacuum distillation of the heavy product.

The run was stopped when enough coal liquid was produced for the 25 L of distillate required by the contract.

Table 5.1

OPERATING CONDITIONS

Feed Slurry	
Rate (Kg/hr)	5.06
Solvent/maf fresh coal	2.5/1
Feed Gas	
Rate (L/hr)	1161
(L/Kg maf coal)	406
Temperatures (°C)	
Slurry Preheater	295
Gas Preheater	350
Reactor	410
Gas Separator (M3X1)	200
Pressure (MPa)	24.2
LHSV (h^{-1})	0.97

5.1 MATERIAL BALANCE CALCULATIONS

The material balance calculated for this run is shown in Fig. 5.2

Gas Stream Calculation

The D/P cell used for feed gas flow measurement was calibrated before the run. The flow of the feed gas was calculated using the calibration curves and the flow data recorded. All product gas streams were analyzed for

$$H_2,\ CO,\ CO_2,\ CH_4,\ C_2H_6,\ C_3H_8,\ H_2S.$$

The chromatograph data were calculated using external and internal normalization methods and results were checked for consistency. Molecular weights of the gases were calculated from the G.C. analysis for flow, and volumes were plotted against time. The total feed volume of each stream was obtained from the volume versus time curves and total weights were obtained using the G.C. analysis.

Non-Gaseous Calculation

The weight of feed slurry and the weights of slurry components were calculated separately for each batch.

Weights of feed as obtained directly from the plot of load cell readings versus time were corrected for water lost by evaporation from the feed vessel, samples and pump leaks.

The total weight of heavy product was obtained from the plot of heavy product cummulative weight, collected in M4X5 and M4X6, versus time. The composition of the heavy product was calculated using average values of the % THF insolubles and % H_2O analysis of samples taken from M3X1.

For conversion calculations, the net total weights of the following components in the heavy product were calculated: Water, THF insoluble content, THF soluble organic.

The total amount of light product collected in M3X3 was weighed and separated in water and organic liquid.

The mass balance calculated for this run was 97%.

5.2 RECYCLE CALCULATIONS

The solvent recycle procedure adopted was to collect the heavy oil produced over a period of 4 - 5 hours (18-20 Kg) and subject the composite to vacuum distillation to recover the 250-420°C fraction. The other products of distillation, collected and weighed, were the -250°C fraction and the distillation residue. For logistic reasons the distillation was stopped at 420° equivalent ABP, leaving some distillable material in the residue to keep its fluidity. The water was collected in the -250°C fraction and was

separated visually from the organic liquid.

Four distillations were carried out during the run for recycle solvent recovery. The recycle solvent was a combination of the distillate 250 - 420°C fraction mixed with M4X5 heavy oil which was used without any treatment. The ratio between these two components was adjusted in such a way that the coal slurry did not exceed 37% solids content.

5.3 PREPARATION OF THE -350°C FRACTION

Approximately 36 Kg of the liquid product obtained in the last 10 hours of the run was distilled under vacuum into three fractions: a -205°C fraction which was mostly water, a 200 - 350°C fraction which was analyzed and shipped to CANMET ERL and a 350 - EBP remaining fraction which was mixed in the stand-by start-up solvent.

The remaining product of this run was distilled for the preparation of start-up solvent for the next run.

The pitch from all distillation was stored as is. In addition to the 21 L of the 200 - 350°C fraction, a combination of light distillate oil collected during the run, mostly -250°C fraction from distillations was obtained, analyzed and 4 litres were shipped to CANMET as "naphtha" fraction, following a discussion with the Scientific Authority.

5.4 COAL AND SOLVENT CHARACTERIZATION

A homogeneous representative sample (1 Kg) of coal was obtained and proximate and ultimate analysis was carried out, the results of the analyses are shown in Table 5.2.

The start-up solvent was the 230 - 430°C fraction obtained by the vacuum distillation of 59 Kg of lignite-derived liquid product of Sandwell Technologies runs 513 and 514.

A representative homogeneous sample of the starter solvent was analyzed for elemental composition and simulated distillation.

Table 5.3 presents the analyses of the start-up solvent. The distillation curve of the start-up solvent is shown in Figure 5.1. 2 litres of this material were shipped to CANMET ERL with the final product.

Table 5.2

ULTIMATE AND PROXIMATE ANALYSIS OF THE COAL

Proximate Analysis	
(% weight)	
Moisture (coal a received)	24.5
Ash (mf coal)	15.0
Volatile (mf coal)	39.3
Fixed Carbon (mf coal)	45.7
Ultimate Analysis	
(% weight maf coal)	
C	69.4
H	4.8
N	1.3
S	0.9
O (by difference)	23.6

Table 5.3

ELEMENTAL ANALYSIS AND SIMULATED DISTILLATION

OF START-UP SOLVENT

Elemental Analysis

C	90.00
H	6.80
N	1.00
S	0.25
O (by difference)	1.95

Simulated Distillation

Weight % distilled	Temperature (^{o}C)
IBP	186.9
10	241.9
20	261.8
30	276.4
40	289.6
50	303.8
60	308.4
70	324.4
80	341.7
90	349.9
FBP	466.1

5.5 DISTILLATE PRODUCT CHARACTERIZATION

The two fractions of lignite-derived distillate were analyzed for simulated distillation, elemental composition and densi y.

The results are presented in Table 5.4. The densities are 0.9335 and 1.0335 for the -250°C and -350°C fractions respectively. The values which are higher than those of similar distillation range petroleum products suggest highly aromatic nature of the coal-derived liquids, as it is also indicated by the H/C rations in the elemental analyis.

The simulated distillation of the two fractions shows that the last 20% in the case of the "naphtha" and 10% in the case of the -350°C fraction boil above the desired limits, due probably to the poor eficacity of the packed column used for the fractionation.

Table 5.4

DISTILLATE FRACTIONS ANALYSIS

	-250°C Fraction	205-350°C Fraction
Density (g/cc at 21°C)	0.9335	1.0335
Elemental Analysis (% weight)		
C	87.00	89.40
H	9.50	6.55
N	0.65	0.85
S	0.27	0.25
O (by difference)	2.85	2.95
H/C atomic	1.31	0.88
Simulated Distillation		
% weight distilled	Temperature (°C)	
IBP	77.2	99.5
10	143.2	208.5
20	168.0	234.5
30	183.3	254.8
40	196.1	267.9
50	204.8	280.1
60	213.3	292.3
70	227.7	305.8
80	245.5	313.1
90	297.8	333.2
FBP	497.6	471.8

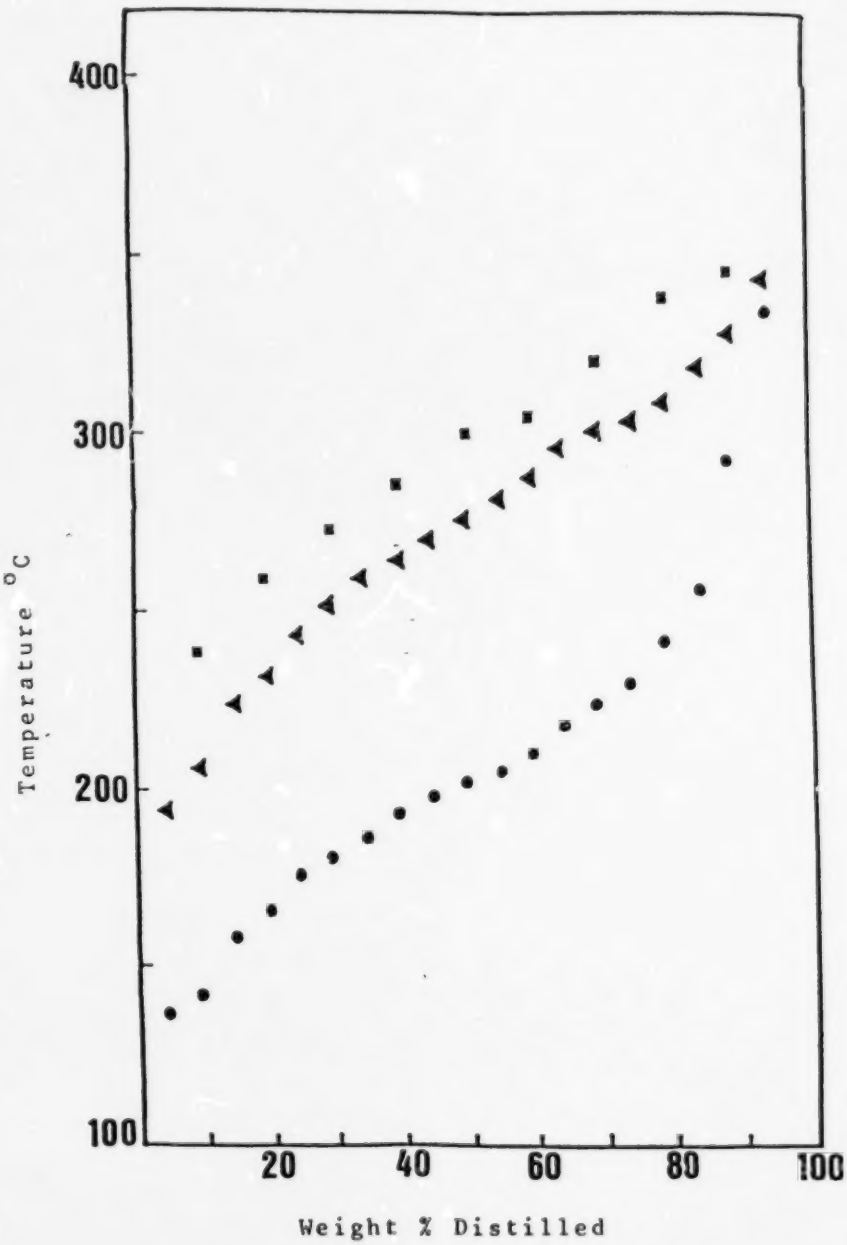

Figure 4.1 Distillation Curves

■ Start-Up solvent

▲ 205-350°C Distillate product

● -250°C Distillate product

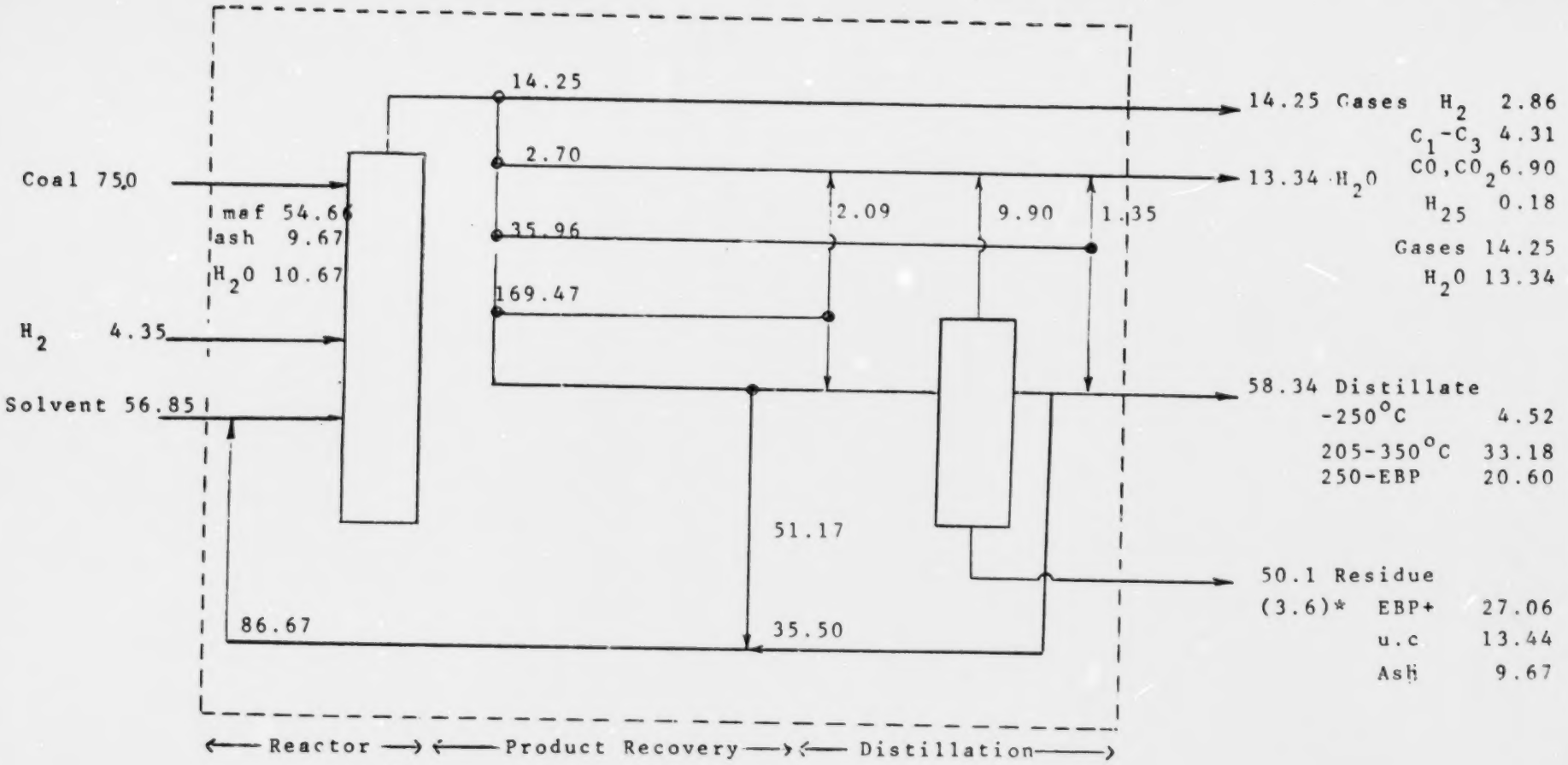

Fig. 5.1 RUN 515 MATERIAL BALANCE

* Estimated losses on the still

5.6 Coal Conversion Calculation

The conversion of coal was calculated by considering the material balance over the PDU, as shown by the inner dotted line in Figure 5.2, and using the following assumptions:

1. Ash (in coal) in equals ash out.

2. The THF insolubles in the recycle feed stream remain unchanged at the outlet.

$$\text{Coal conversion} = \frac{C_1 - (F_2 - A_1 - F_1)}{C_1} \times 100$$

C_1 = Total Kg of maf coal in
A_1 = Total Kg of ash in
F_2 = Total Kg of THF insolubles in product
F_1 = Total Kg of THF insolubles in the recycle feed stream.

From Figure 5.2 the coal conversion is 75.6%